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INTERNAL FRICTION AND MODULUS STUDIES ON AUSTEMPERED DUCTILE IRON

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13. ABSTRACT (Maximum 200 words)

Internal friction and modulus measurements were performed on three difference compositions of austempered ductile iron. These measurements were performed using a TA Instruments' Dynamic Mechanical Analyzer under a variety of thermal cycling conditions. The internal friction data exhibit the Snoek effect peaks at about 250°C. Using peak positions measured at different frequencies, the activation energy of the process was measured and found to average 31.9 kcal/mole. This is consistent with the activation energy of the diffusion of carbon in γ-Fe (32 kcal/mole). The untransformed austenite was decomposed by holding at 400°C. After decomposition, the internal friction peak disappeared and the modulus increased 10 percent. These results confirm that the internal friction and reduced modulus arise from anelastic effects in the untransformed austenite phase.

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INTRODUCTION

Austempered ductile iron (ADI) is cast as conventional nodular iron. The carbon in this type of iron forms into spheroids of graphite during solidification. The standard heat treatment for ADI involves austenitizing followed by a quench and hold at an austempering temperature for a time that yields optimum properties. A bainitic-type reaction takes place during austempering. As the bainite reaction proceeds, carbon partitions from the ferrite region into the remaining austenite. This untransformed austenite decomposes to ferrite and carbide at 400°C and above. The percentage of untransformed austenite is a strong function of austenitization temperature, austempering temperature, and composition. For this study we used the compositions and heat treatments listed in Tables 1 and 2.

A properly optimized austempered ductile iron material is reported to exhibit a good combination of strength and ductility (refs 1-4). Toughness measurements of ductile irons using unnotched specimens have been found to give good results under static conditions (refs 2-4). It is known that ADI has good dumping properties which were assumed to be derived from the spheroidal graphite nodules intermixed in the ausferrite matrix (refs 5,6). This study of modulus and internal friction provides new information on the inherent damping properties of ADI.

Internal friction measurements provide useful information about the structure and composition of a material (refs 1-9). The purpose of this report is to present the results of these measurements on ADI. The internal friction and modulus measurements were performed on the TA Instruments' Dynamic Mechanical Analyzers (DMA), models 982 and 983. The activation energy of the internal friction process was obtained from the frequency dependence of the internal friction peak. Changes in internal friction peak amplitude during an isothermal hold were used to measure the carbon precipitation in the untransformed austenite.

Internal friction relates to the ability of a material to absorb vibrational energy (ref 10). One mechanism for vibrational energy absorption arises from the presence of a solute in a metallic solid solution. Interstitial positions can alternately enlarge and contract during each cycle in a vibrating system. Solute atoms respond to the strain pattern by jumping from contracted to expanded interstitial sites. For example, in dilute iron-carbon alloys, this occurs along the $\langle 100 \rangle$ direction. This stress-induced atomic motion is accompanied by an energy loss. For a given carbon concentration, the energy loss reaches a maximum when the vibrational frequency of the metal approaches the carbon atoms' normal atomic jump rate. For example, this maximum energy loss appears at a 1 cycle/sec vibration rate in iron at room temperature. The energy loss of an iron sample thus vibrated is found to be proportional to the carbon in solid solution. This technique has been used to obtain the carbon concentration in iron ferrite (ref 8). Internal friction can, therefore, provide valuable information about solute distribution, alloy structure, precipitation effects, and diffusivity in some cases.

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SPECIMEN PREPARATION

The DMA specimens were machined from ADI material labeled T158, G1, and G3 into rectangular strips, 0.95 to 1 mm thick and 9.5 to 11.5 mm wide. The length of the specimen is the distance between the clamps, which ranged from 20 mm to 55 mm. The materials were cast using the chemistry requirements in Table 1 and heat treated as described in Table 2.

EXPERIMENTAL METHOD

The DMA 982 and 983 operate by forcing a constant amplitude oscillation at resonant or user-selected frequency. The sample is flexed by two parallel arms oscillated by an electromagnetic driver at a user-selected amplitude. The sample's viscoelastic properties determine the resonant frequency of the system. As these properties change with temperature and time, the modules adjust the electric energy to maintain the selected amplitude as the resonant frequency varies. The DMAs measure the frequency of the oscillation (which relates to the elastic modulus) and the electrical energy necessary to maintain the oscillation amplitude (which relates to the material's inherent damping). These values are used by the TA analysis software to calculate E' (tensile storage modulus), E" (tensile loss modulus), G' (shear storage modulus), G" (shear loss modulus), and Tan Delta (loss factor, E"/E'), using the equations and variables found in the Appendix.

In fixed frequency mode, the DMA 983 forces the sample to oscillate at a user-defined frequency using a sinusoidal driver signal. The lag between the driver signal and the LVDT-monitored sample displacement is the phase angle. The phase angle and drive signal are the variables used to calculate the properties.

The DMA samples were positioned perpendicular to clamps, carefully tightened to 18 ft-lbs by alternating tightening of the clamps in 2 ft-lb steps. The system operated in an inert atmosphere of helium at a flow rate between 1750 and 2150 cc/min in the DMA 982 and between 1300 cc/min and 1750 cc/min in the DMA 983 to maintain a comparable flow rate at the gas outlet. After a twenty-minute purge, the samples were ramped up at 5°C/minute to the set point (usually 300°C). In a half dozen experiments, the samples were held at 400°C for 24 hours, to study the effect of decomposition of the untransformed austenite on modulus and internal friction. The raw data was analyzed by the TA DMA analysis software and converted to ASCII for spreadsheet analysis.

RESULTS AND DISCUSSION

The internal friction data exhibit Snoek peaks (Figures 1 and 2). The peak positions were determined by fitting the upper 10 percent of the measured peaks to a quadratic

equation.[1] The internal friction, as represented by Tan δ, is a function of frequency, ν, and relaxation time, τ, where

$$\tau = e^{-H/RT} \tag{1}$$

and H = activation energy, and R = universal gas constant = 2 cal/mole/°K.

If Tan δ versus 1/Temperature curves are obtained for different frequencies, they can be superimposed by a horizontal shift, since Tan δ is a function of $\nu\tau$ and τ is an Arrhenius equation. Thus, the positions (in temperature) of the Snoek peaks exhibited by the internal friction data and the frequency of the peaks can be related by (ref 9)

$$\ln(f_2/f_1) = -(H/R)*\Delta(1/T) \tag{2}$$

Plotting 1/$Temp_{abs.}$ against the ln(freq.) gives a straight line with a slope of -H/R. The slopes of these graphs were obtained by linear regression. Figures 3 and 4 show results for internal friction measurements of ADI performed in the resonant mode on the DMA 982 and in the fixed frequency mode on the DMA 983, respectively.

The resonant mode internal friction measurements were performed on three T158 specimens on the DMA 982 over a frequency range of 10 to 30 Hz. The peak positions were analyzed and plotted against the natural log of the frequency as described above. The activation energy was calculated to be 29.5 kcal/mole. The fixed frequency mode internal friction measurements were performed on the DMA 983 and G3 samples over a wider frequency range (0.01 to 20 Hz). The activation energy was calculated to be 34.4 kcal/mole.

The activation energy values indicate that the Snoek peaks arise from diffusion of carbon in a face-centered-cubic γ-Fe. The published activation energy value for the diffusion of carbon in γ-Fe is 32 kcal/mole, and the value for diffusion of carbon in α-Fe is 18 kcal/mole (ref 9). The average of the activation energies by the two modes is 31.9 kcal/mole, which corresponds to the value for γ-Fe. In ADI the body-centered-cubic ferrite (α-Fe) contains a negligible level of carbon, and the untransformed austenite (γ-Fe) possesses a large percentage of carbon, 1.5 to 2 percent, so that the effect can only arise from the untransformed austenite.

Internal friction and modulus measurements were also made during twenty-four hour isothermal hold experiments at 400°C. Under these conditions, the untransformed austenite decomposes to bainite, which is a mixture of ferrite and carbides. In 300°C runs performed

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[1] The raw data had been smoothed by the TA data analysis software. The curve fitting was performed by Jandel's TableCurve version 3.10. The first derivatives of the quadratic equation were solved for peak positions.

after the 400°C twenty-four hour runs, the Snoek peaks completely disappear (Figures 1 and 2), and the internal friction values are all lowered to the "baseline" values.[2]

Snoek peak amplitudes for carbon in iron are proportional to the carbon content (refs 7,8). Thus, the decline in the internal friction during the isothermal hold is a measure of the precipitation of the carbon. This percentage of carbon precipitating out of the untransformed austenite can be found using the internal friction values and the equation

$$W = (Q_0^{-1} - Q_t^{-1})/(Q_0^{-1} - Q_f^{-1}) \tag{3}$$

where

$$Q_0 = \textit{starting internal friction value}$$

$$Q_t = \textit{internal friction value at time } t$$

$$Q_f = \textit{final internal friction value}$$

The results of these calculations during the isothermal holds are shown in Figures 5 and 6. The untransformed austenite decomposes into ferrite and carbides.

The modulus showed the same asymptotically changing values during the twenty-four hour hold at 400°C. By the end of the twenty-four hour hold at 400°C, the room temperature modulus values had increased by approximately 10 percent, and the 400°C values increased by 13 percent. The initial decrease in modulus on heating to 400°C is 15 percent before decomposition and 7.5 percent after decomposition. An inflection in the modulus-temperature curve occurs at approximately 250°C. This break corresponds to the Snoek peak position in the internal friction. It disappears, like the Snoek peak, after decomposition. The presence of free graphite nodules has been considered as the cause of the lower modulus values typical of ADI (ref 6). However, as demonstrated in this report, the untransformed austenite accounts for the lowered values.[3]

CONCLUSIONS

The dominant contribution of the untransformed austenite in ADI to internal friction has been demonstrated. The activation energy of the process generating the internal friction

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[2] A previously performed gunsteel internal friction measurement is given in the Appendix. Its maximum value at 400°C of approximately $8e^{-4}$ compares to the after-decomposition results. Since steel has a negligible internal friction signal, this supports the conclusion that the internal friction values and good inherent damping properties measured were derived from the untransformed austenite.

[3] Previous measurements performed on gunsteel show a modulus range of 200 to 210 GPa.

peaks in ADI was measured and found to be consistent with the Snoek effect of carbon in γ-Fe (untransformed austenite). Therefore, these peaks are associated with the diffusion of carbon in γ-Fe. The internal friction peaks were diminished to baseline values after the decomposition of the untransformed austenite. Similarly, the modulus experienced a significant increase in value and decrease in temperature dependence with decomposition of untransformed austenite. Internal friction and modulus measurements provide a useful alternative method for ADI transformation behavior analysis.

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Table 1. Chemical Composition of the ADI Materials

Material	C	Si	Mn	P	S	Ni	Cr	Mo	Mg	Cu	Al	B	Ti	Sn
G3 & G1	3.71 ± 0.11	2.72 ± 0.07	0.27 ± 0.01	0.009 ± 0.002	0.957 ± 0.072	0.0046 ± 0.0014	0.032 ± 0.004	0.27 ± 0.01	0.057 ± 0.004	0.870 ± 0.073	0.0151 ± 0.0016	0.0002 ± 0.0002	0.0046 ± 0.0014	0.0011 ± 0.0006
T158	3.2 to 3.8	2.45 to 2.65	0.3 max	0.03 max	0.015 max	1.2 to 1.5	resid (0.051 to 0.04)	resid (0.003 to 0.007)	aim 0.035 (0.053 to 0.04)	resid				

Table 2. Heat Treatment Procedures

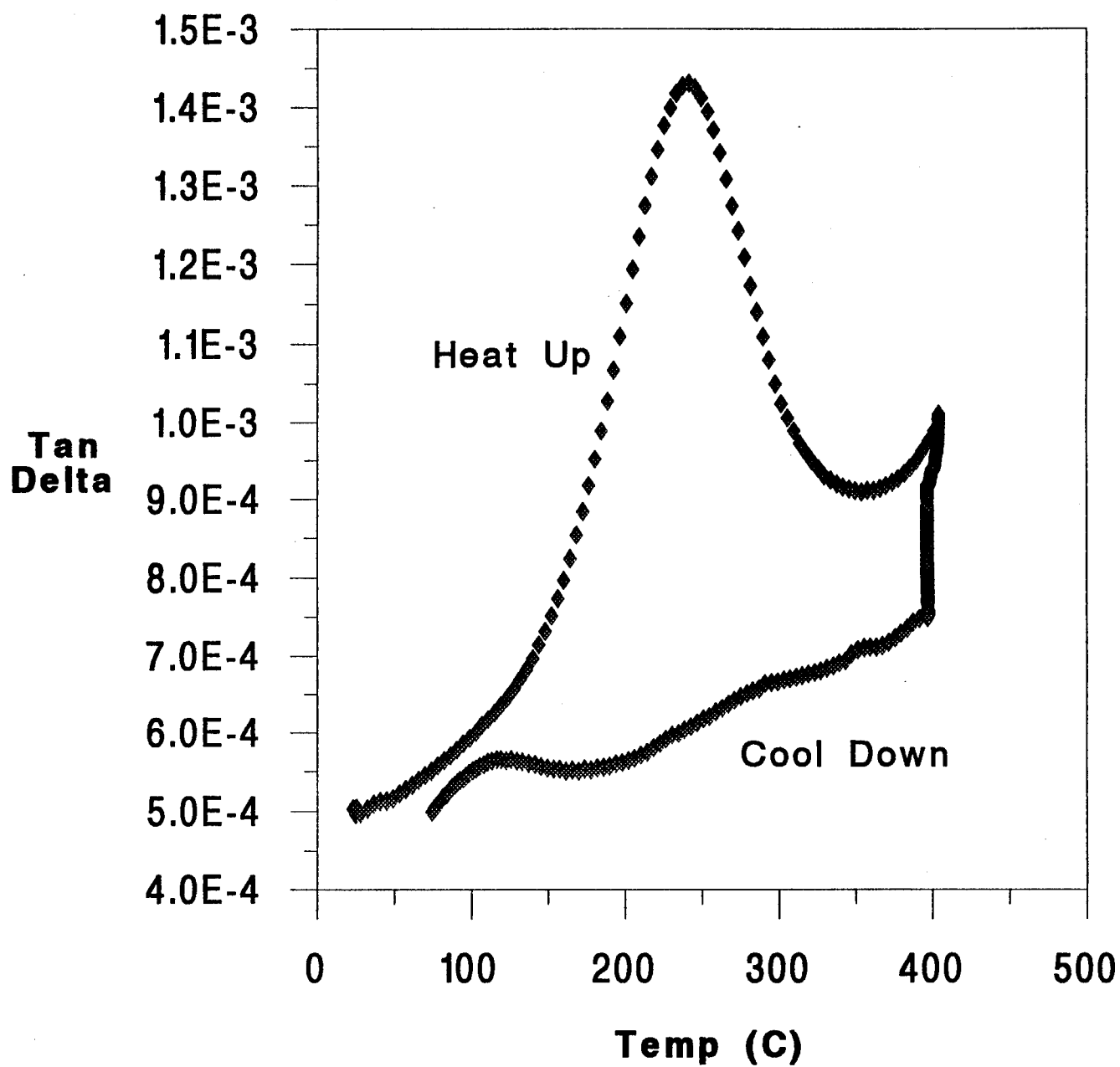| Material	Stress Relief/Preheat		Austenitizing Parameters		Austemper Parameters		Quench Medium
	Temperature	Time	Temperature	Time	Temperature	Time	
G3	1100°F/593°C	120 mins	1625°F/885°C	150 mins	600°F/316°C	150 mins	Molten Salt
G1	1100°F/593°C	105 mins	1635°F/891°C	100 mins	620°F/327°C	100 mins	Molten Salt
T158			1650°F/900°C	1.25 hrs	625°F/330°C	1.75 hrs	Water

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Figure 1. A T158 ADI sample held at 400°C for 24 hours to decompose the untransformed austenite, exhibiting a peak that does not return after the decomposition.
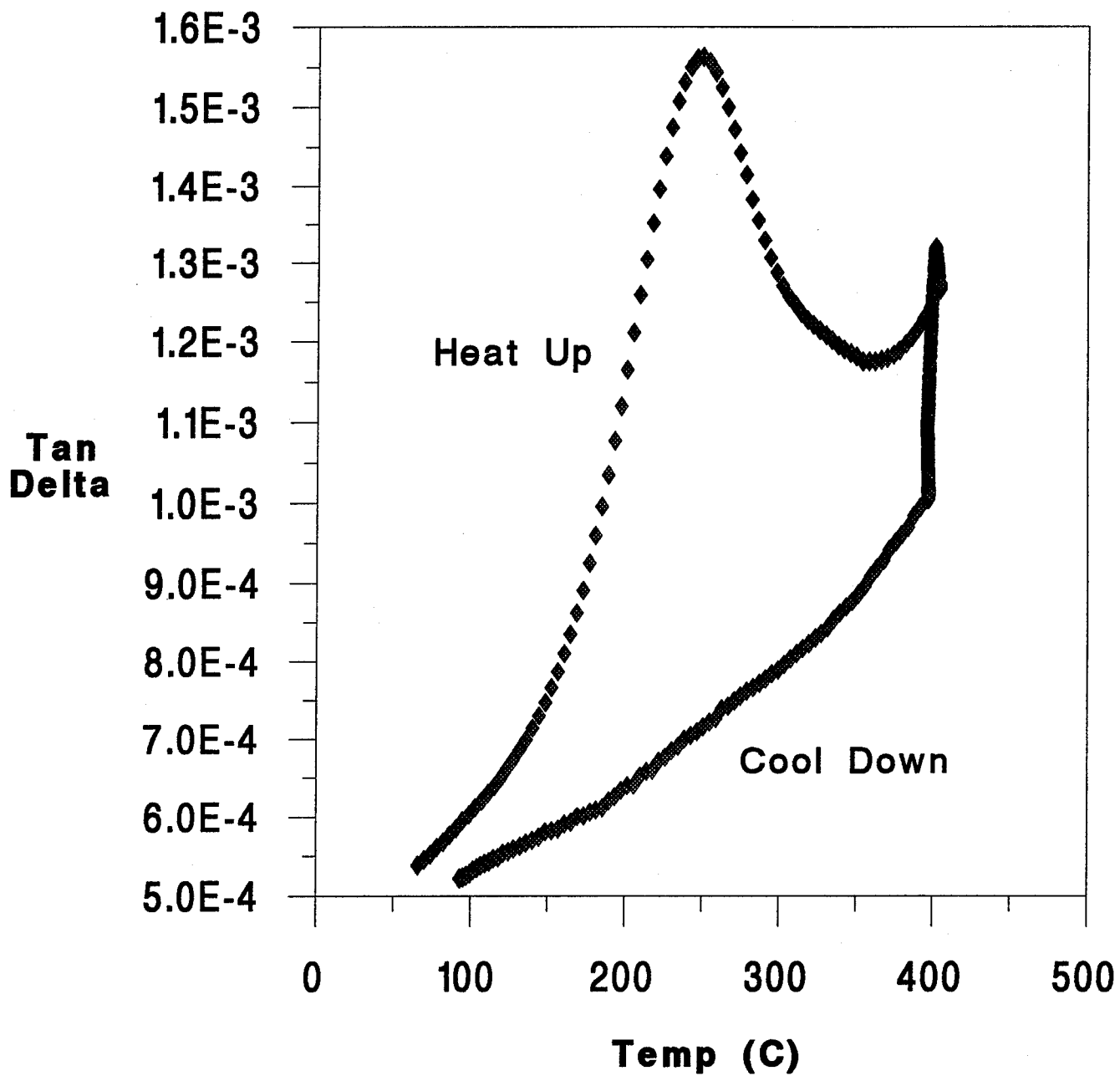
Figure 2. A G3 ADI sample held at 400°C for 24 hours to decompose the untransformed austenite, exhibiting a peak that does not return after the decomposition.
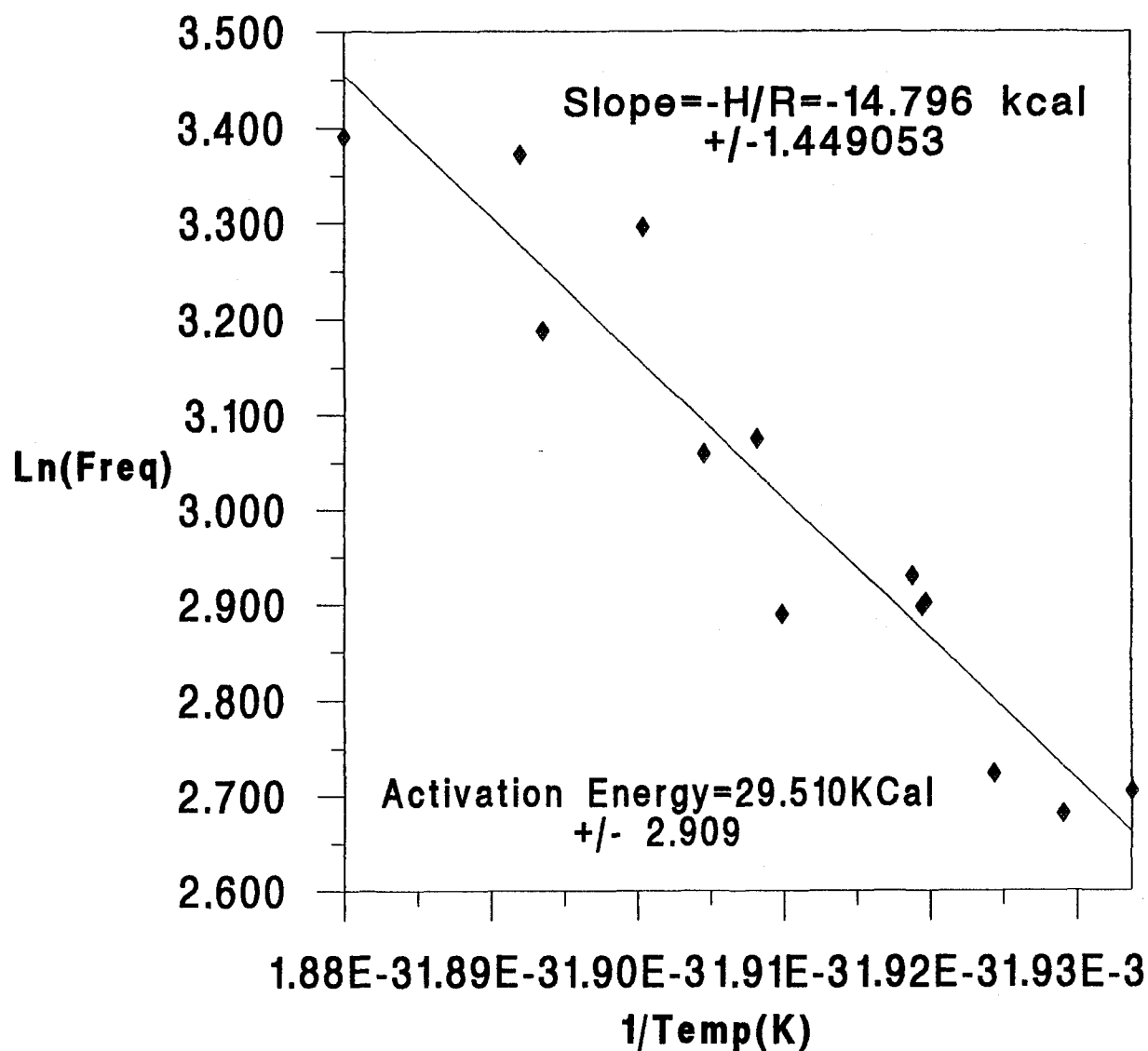
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Figure 3. Data points representing the Snoek peak temperature positions for the resonant frequencies of the three T158 samples clamped at various lengths in the DMA 982. The activation energy equals the slope of the linear regression line multiplied by -R (2 cal/mole).

Figure 4. Data points representing the Snoek peak temperature positions for the resonant frequencies of the G3 samples clamped at various lengths in the DMA 983. The activation energy equals the slope of the linear regression line multiplied by -R (2 cal/mole).

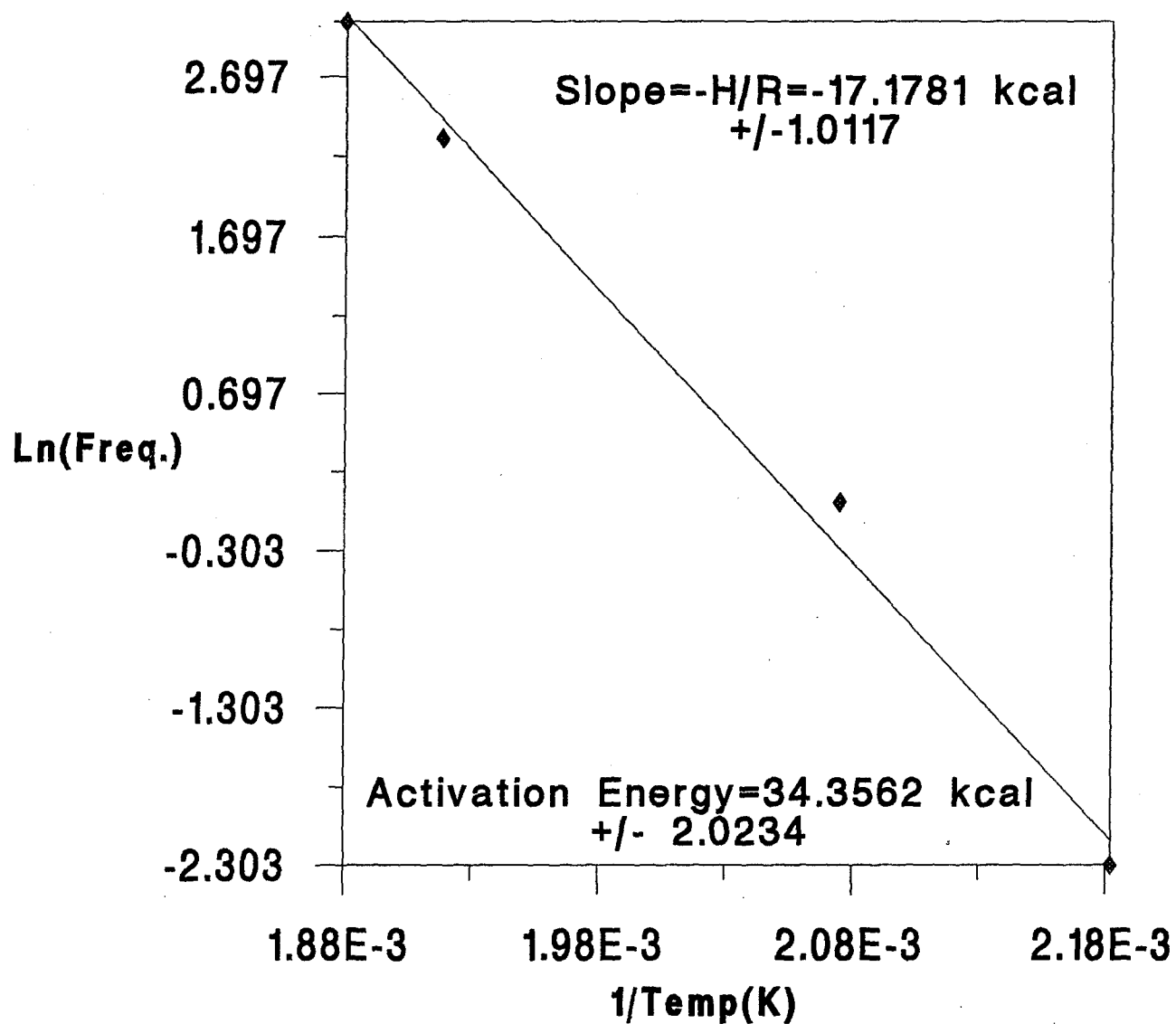Figure 5. The internal friction value, Tan Delta, of T158 ADI declining asymptotically during a 24-hour isothermal hold at 400°C. The percentage of precipitation calculated from these values is also shown for the 24-hour period.
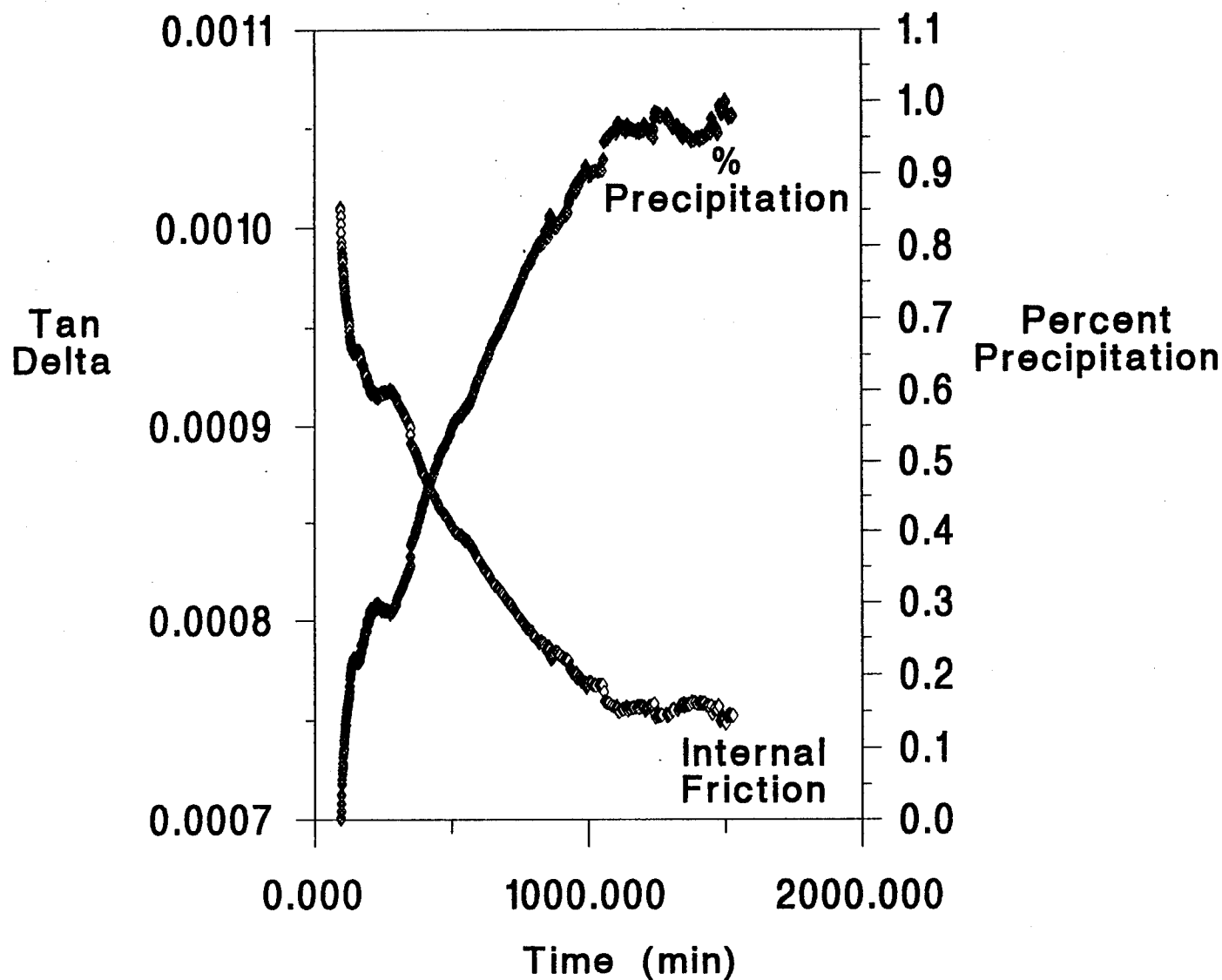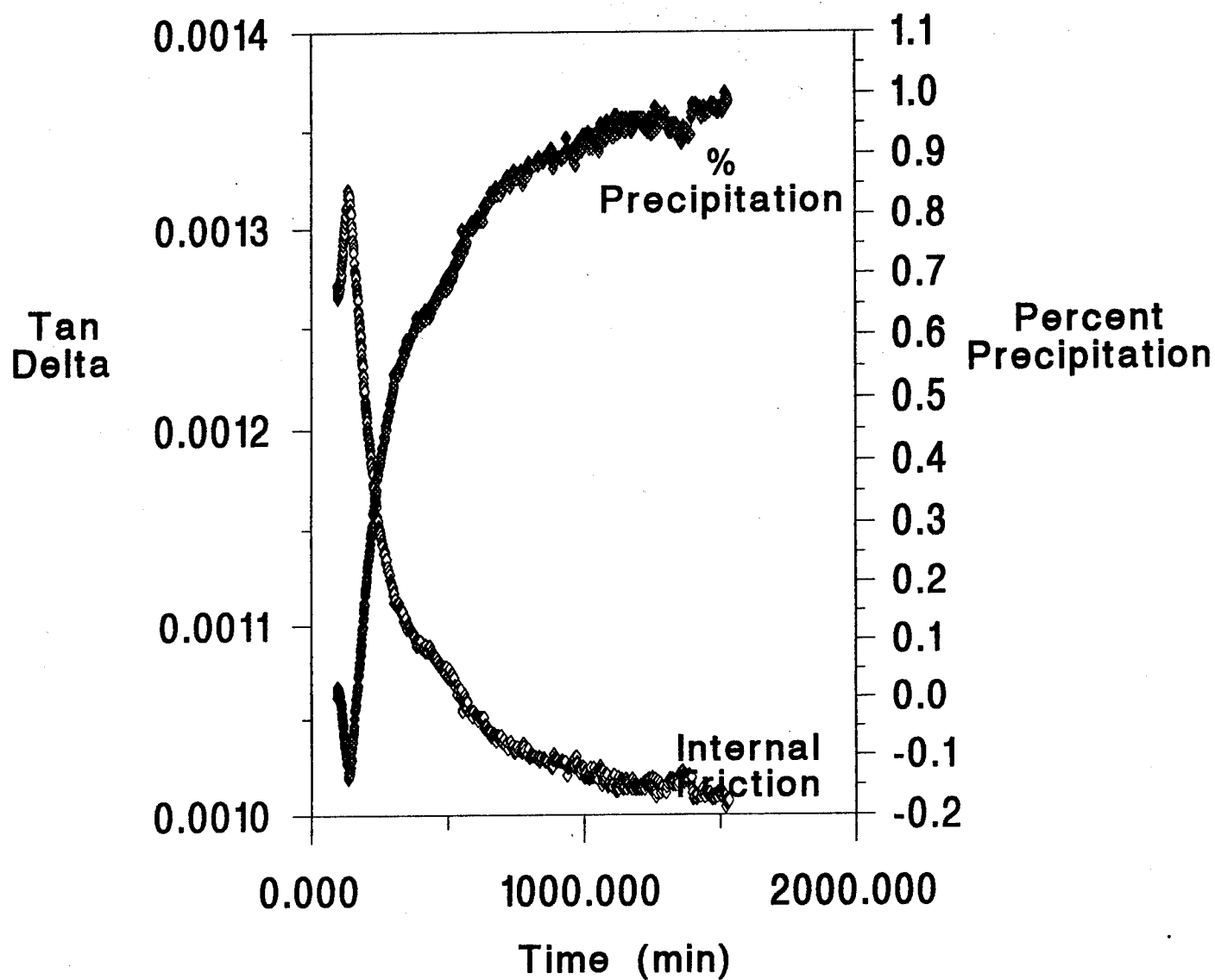
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Figure 6. The internal friction value, Tan Delta, of G3 ADI declining asymptotically during a 24-hour isothermal hold at 400°C. The percentage of precipitation calculated from these values is also shown for the 24-hour period.

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APPENDIX
EQUATIONS FOR THE DMA 982 & 983

Measured Signals:

V	=	Drive signal (mV)
f	=	Frequency (Hz)
δ	=	Phase angle (radians)
δ	=	Measured signal for fixed frequency mode
δ	=	$\pi/2$ for resonance mode

Experimental Parameters:

T	=	Thickness of rectangular sample (mm)
W	=	Width of rectangular sample (mm)
L	=	Length of sample (mm)
r	=	Radius of cylindrical sample (mm)
a	=	Oscillation amplitude (mm)

Sample Constants:

ΔL	=	Length correction constant (mm)
σ	=	Poison's ratio
α	=	Shear distortion factor

Instrument Constants:

D	=	Clamping distance (mm)
R	=	Distance from flexure pivot center to pivot at which a is measured in instrument calibration (127.85 mm)
J	=	Single arm inertial moment (kg m^2)
C'	=	Drive signal constant (mm/(mV sec^2))
K'	=	Instrument parallel storage stiffness (N m)
K''	=	Instrument parallel loss stiffness (N m)= $K_n''f/f_n$
f_n	=	Frequency at which K_n' was measured (Hz)
f_m	=	Frequency at which J_{Cm}'' was measured (Hz)
f_∞	=	Maximum frequency obtained with an infinitely stiff sample (Hz)

Calculated Intermediate Values:

L'	=	Effective sample length (mm) = measured L + ΔL
B	=	Distance between pivot centers (mm) = sample length + 2(D)
A	=	Cross-sectional area of sample (mm^2):
		T x W for rectangular samples
		πr^2 for cylindrical samples
I	=	Geometrical inertial moment of sample cross section (mm^4):
		$T^3W/12$ for rectangular samples
		$\pi r^4/4$ for cylindrical samples

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$$\omega = 2\pi f$$

$$M_0 = \left(\frac{2J}{R}\right)4\pi^2 C'V$$

$$\theta_0 = \frac{a}{R}$$

$$\beta = \alpha + \frac{L'^2 A}{24(1 + \alpha)I} + \frac{A}{L'}(J_c{'}\ G' + J_c{''}\ G'')$$

$$\gamma = (A/L')(J_c{'}\ G'' - J_c{''}\ G')$$

$$K^2 = \frac{M_0\cos\delta}{2J\theta_0} + \omega^2$$

$$(wd) = \frac{M_0}{2J\theta_0}$$

Primary Equations:

Shear Storage Modulus:

$$G' = (2Jk^2 - 2K')\frac{\beta^2 + \gamma^2}{\beta}\ \frac{L'}{B^2 A} - \frac{\gamma}{\beta}G''$$

Shear Loss Modulus:

$$G'' = [2J(wd) - 2K'']\frac{\beta^2 + \gamma^2}{\beta}\ \frac{L'}{B^2 A} + \frac{\gamma}{\beta}G''$$

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Flexural Storage Modulus:

$$E' = 2(1+\sigma)G'$$

Flexural Loss Modulus:

$$E'' = 2(1+\sigma)G''$$

Tan Delta:

$$G''/G' \quad or \quad E''/E'$$

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